

Report on Standard Solutions

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Last year the General Referee recommended discontinuance of the two remaining outstanding topics, and continued study and evaluation of this chapter with a view toward expanded coverage.

The study has been completed and the entire 10th edition of *Official Methods of Analysis* has been reviewed.

It is recommended that the title of Chapter 42 be changed from "Standard Solutions" to "Standard Solutions and Materials"; that the chapter coverage

be expanded to include metal, non-metal, and organic reference materials which are common to two or more current official methods, and which are not primary standards of themselves; and that Associate Referees be appointed in these general areas to explore and identify appropriate secondary standard reference materials and devise appropriate analytical methods to determine purity and other factors as compared to available primary standards or other absolutes.

Recommendations

The recommendations of the Referee are those given in the report of Subcommittee A; see *JAOAC* 53, 379 (1970).

Report on Tobacco

By C. L. OGG (Eastern Utilization Research and Development Division, U.S. Department of Agriculture, 600 E. Mermaid Lane, Philadelphia, Pa. 19118)

Two collaborative studies were conducted during the past year in cooperation with the Analytical Methods Committee of the Tobacco Chemists Research Conference. One study was a continuation of the work on a method for determining humectants in tobacco products, and the other dealt with tar and nicotine determination in cigarette smoke.

Nine collaborators analyzed three pairs of samples for propylene glycol, glycerine, and triethylene glycol, using the gas chromatographic procedure developed by the Associate Referee. The results were considerably improved over those of previous studies; coefficients of variation for propylene glycol and glycerine were reduced to less than half the values of the last study.

Because of significant systematic errors for samples with high glycerine and low triethylene glycol content and fairly high *F* values for three other samples, the Referee does not concur with the Associate Referee's recommendation that the method be adopted as official first action. Further study is recommended to try to find a better internal standard.

The method for tar and nicotine used in this year's collaborative study differed from the method used in the previous studies by the inclusion of a procedure for determining the moisture in the particulate matter. Ten collaborators reported tar and nicotine values for 200 monitor cigarettes each, 40 ports (samples) of 5 cigarettes each.

Ten collaborators also smoked 60 cigarettes (12 ports) each of 10 samples constituting 5 pairs. Samples were randomized as to port and the smoking was distributed over 6 days, 3 during one week and 3 the next. The two studies provided over 1500 values for both tar and nicotine. Continued study is recommended to try to determine the cause for some divergence of values for cigarettes with high tar and nicotine content.

The official first action colorimetric and gas chromatographic methods for menthol in tobacco,

JAOAC 51, 650-653 (1968), have been in use for two years with no adverse reports. It is therefore recommended that both methods be adopted as official final action.

Recommendations

The recommendations of the Referee are those given in the report of Subcommittee A; see *JAOAC* 53, 379 (1970).

GENERAL REFEREE REPORTS: SUBCOMMITTEE B

Report on Antibiotics

By WILLIAM W. WRIGHT (Division of Pharmaceutical Sciences,
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This report describes our progress in developing and validating methods of assaying for antibiotics in animal feeds. During the past year two collaborative assays were completed and plans were made for five more collaborative studies.

The Associate Referee on Bacitracin in Feeds has not been able to submit to collaborative study the improved method involving a better feed cleanup and a more sensitive assay procedure, *JAOAC* 52, 681-685 (1969). In accordance with the General Referee's direction, the Associate Referee has tried the procedures on a number of feeds of varied composition. These investigations have led to the conclusion that the improved method is likely to have general applicability. A collaborative assay is being planned.

The Associate Referee on Erythromycin Thiocyanate has investigated the effect of sample size in the method recommended last year for collaborative study, *JAOAC* 52, 672-675 (1969). Satisfactory precision is obtained on sample sizes ranging from 10 to 100 g, provided a 10-fold ratio of solvent to sample is used. He has also found that the method eliminates the problem previously encountered due to binding of erythromycin to bentonite in some feeds. A collaborative assay is planned.

A new Associate Referee on Monensin in feed has been appointed. He has developed a method of sample cleanup and extraction, *JAOAC* 53, 49-53 (1970), in which the feed is placed in a chromatographic column over alumina. The column is eluted with 90% methanol and the eluate is diluted with methanol-water (1 + 1). The final solution is assayed by a conventional *B. subtilis* cylinder plate assay. It is recommended that the assay be submitted to collaborative study.

The collaborative study of the assay method, *JAOAC* 53, 60-68 (1970), for neomycin in feeds has been completed. Eighteen collaborators applied the method to 24 feed mixes comprised of four di-

verse feed types fortified at 6 levels of neomycin content each. An average neomycin recovery of 95.5% was obtained. It was found, as expected from preliminary investigations, that low neomycin recovery occurred with the feed containing 65% soybean meal. When that feed was excluded from the statistical evaluation, an average recovery of 100.3% was obtained, with the standard error of the mean being 0.8%. The Associate Referee has recommended that the method be adopted as official first action and the General Referee concurs.

The Associate Referee on Nystatin is planning a collaborative study of the improved assay method described last year, *JAOAC* 52, 675-678 (1969).

The Associate Referee on Oxytetracycline has surveyed the experience of the official first action method for oxytetracycline in final feeds, *JAOAC* 51, 268, 391 (1968). All laboratories that used the method reported that they had encountered no difficulties, and several commented that it is a definite improvement over the previous method. The Associate Referee has recommended that the method be adopted as official final action and the General Referee concurs.

Collaborative study has been completed on modifications of the official method for streptomycin in feeds, *JAOAC* 53, 54-59 (1970). The results show that satisfactory sample preparation can be achieved using a single extraction instead of three as in the official method. Mechanical shaking and high-speed blending gave equivalent results. Four feed levels ranging from 6 to 75 g/ton were analyzed using sulfadiazine-sensitized *B. subtilis* assay plates, and four levels ranging from 37.5 to 300 g/ton were assayed on unsensitized plates. The results from 18 collaborators showed better results with the sensitized plates. At 37.5 g/ton the average recovery was 95.9% as compared to 87.4% obtained from assays on unsensitized plates. At 75 g/ton the recoveries